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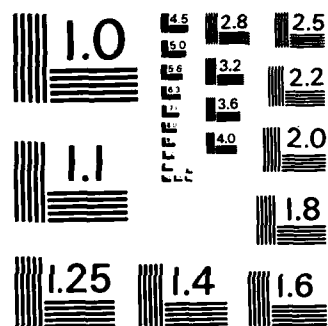
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Bethesda, Maryland 20084



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PAINT BLISTER TEST PROCEDURE

by

D. R. Laster

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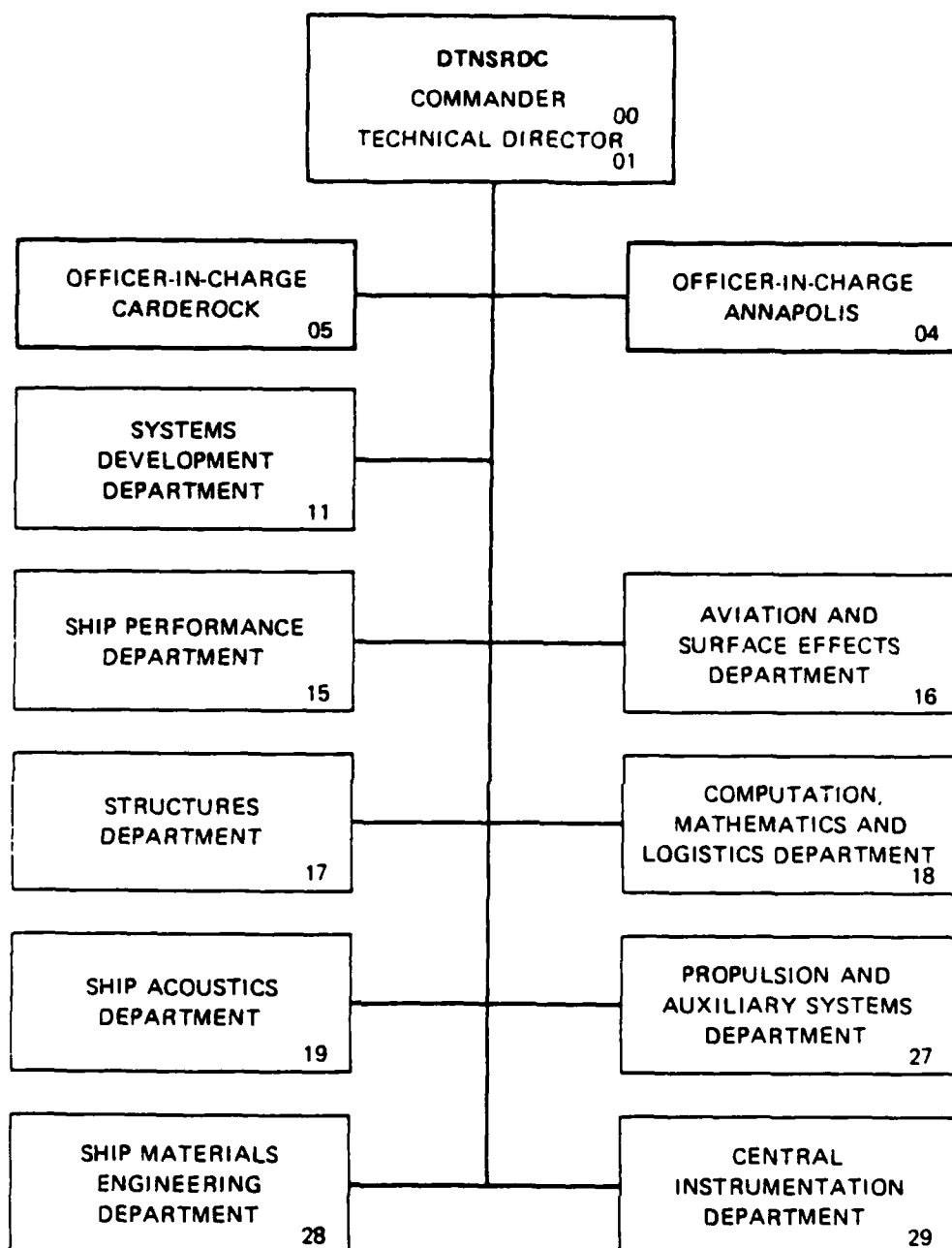
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## REPORT DOCUMENTATION PAGE

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| 19 ABSTRACT (Continue on reverse if necessary and identify by block number)<br><br>A description is given of a paint blister test apparatus and procedure. The test is designed to determine the blister susceptibility of the polyamide epoxy anticorrosive hull paints used in the U.S. Navy. |       |                                                |                                                                                                  |                                                       |                                |
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## ABSTRACT

A description is given of a paint blister test apparatus and procedure. The test is designed to determine the blister susceptibility of the polyamide epoxy anticorrosive hull paints used in the U.S. Navy.

## ADMINISTRATIVE INFORMATION

This work was funded by NAVSEA 05R25, Mr. C. Zanis, through the Surface Ship and Craft Materials Block Program, Program Element 62761N, Task Area SF 61-541-592, Work Unit 1-2803-165-44. The work was performed under the supervision of Mr. H. S. Preiser, Head, Paints and Processes Branch (Code 2841).

## PAINT BLISTER TEST PROCEDURE

### INTRODUCTION

#### OBJECTIVE

The objective of the report is to present a standardized blister test apparatus and procedure that is simple to set up and to operate. The test is for use in determining blister susceptibility of the polyamide epoxy anticorrosive (AC) paints used in the U.S. Navy.

#### CRITICAL TEST ELEMENTS

The test procedure includes four essential test elements that must not be compromised:

(1) Controlled quality substrate: The panel material must be Q-panels\* or their equivalent to prevent substrate material variations from introducing uncertainties and errors. Substrate preparation must be meticulous and consistent.

(2) Controlled, repeatable exposure conditions: The panels are to be exposed in deionized water maintained at 180°F to within 1/2°F with the conductivity held at 20  $\mu$ mhos or less. The panels must not be removed from the water at any time during the test. All inspections are to be made through a window in the exposure tank.

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\*Test panels of quality-controlled steel made by Q-Panel Corporation.

(3) Standardized rating scheme: The blistering of the coatings will be rated using the American Society for Testing and Materials (ASTM) D-714 Pictorial Standards. Additional information or interpolations may be used, but the basic data must include the ASTM D-714 ratings.

(4) Observations of the blistering as a function of time: At least one rating per day must be made on each panel so that the blistering development over the 2-week test period may be determined. It is essential that the person making the ratings do each day's ratings without reviewing the previous day's ratings.

#### BACKGROUND

The Navy is presently seeking anticorrosive hull paints with an effective performance life of 5 to 10 years. The F-150 series polyamide epoxy paints (MIL-P-24441), both as they have been used in the Navy for more than 20 years and as they are presently supplied, cannot meet this life requirement. The original formulation has undergone rather significant modifications in response to environmental and health requirements. It is believed that these modifications have either caused or exacerbated a blister problem. In the investigation of this blistering problem the accelerated laboratory paint blister test reported here was developed to aid in determining differences in blistering modes, tendencies and characteristics of anticorrosive coatings.

The blistering of paints has been a subject of concern to paint formulators, researchers, and users since paints have been in existence. It is one of the more common causes of paint failure. A blister is a defect in the paint-substrate film system that shows up as a raised round or oval patch in the paint film and is locally detached from the surface. Blisters may be wet or dry, may vary in size and distribution, and thus may remain intact within the film or become unstable and rupture to expose the substrate. The substrate can be a metallic or nonmetallic structure or in some cases the intact layer of another paint coating.

Blisters, although they represent a local film failure, are not necessarily devastating. Some blisters grow to a finite size and become stable; others grow, coalesce and ultimately become unstable where they are easily damaged. The damage can be caused mechanically or by water flow in the case of hull paints on a moving ship. There are a variety of causes of paint blistering, some of which are:



- Osmosis: Causative factors are hydrophilic metal contamination, trapped solvents and corrosion products.
- Enhanced H<sub>2</sub>O diffusion: Results from a temperature gradient between substrate and water.
- Cathodic electro-osmosis: The movement of ions through a film to a substrate having a negative charge.
- Cathodic polarization: The migration of cations generally resulting in the generation of hydrogen gas or excess OH<sup>-</sup> ions as part of the cathodic reaction.
- Anodic polarization: The migration of anions resulting in oxide formation of gas generation or anodic corrosion.

Most cases of paint blistering can be explained by the osmotic mechanism.

In the laboratory test described here the osmotic mechanism of paint blistering is enhanced and accelerated by exposing the painted panels to heated, deionized water. The panels are frequently observed so as to develop a time history of the blister development. The relative performance of the paints under test may be ranked with respect to the length of time before the appearance of the initial blisters, the rate at which blister size and/or number grow, or the final pattern of blistering on the test panels at some selected cutoff time.

### PROCEDURES

The specific objective of the blister test described is to compare the inception of blisters and their growth on the various paints under test. The data provided include the blister size and density (rated according to ASTM D-714) and the linear blister resistance, all as functions of time of exposure to the blistering environment. The linear resistance rating is derived from the ASTM D-714 ratings using Table 1.

### PANEL PREPARATION

The substrate selection and preparation can have a major influence on the blister test results and on the repeatability of these results. To ensure that the substrate is uniform for all the panels, steel Q-panels, type "S," with dimensions of 4 in. by 8 in. and thickness of 0.032 in. should be selected for the exposure tests. The panels are solvent-washed in xylene and then grit blasted to a near

white metal finish (SPPC-SP 10, as defined by the Steel Structures Painting Council). The profile must be between 1.5 and 3 mils. After grit blasting the panels are solvent-washed twice in xylene. After adequate air drying the paint to be tested is applied in accordance with the paint manufacturer's instructions. If the panels are not to be used immediately, they must be carefully wrapped in vapor phase inhibitor (VPI) paper.

TABLE 1 - LINEAR BLISTER RESISTANCE RATING MATRIX  
(RATING IS THE SQUARE ROOT OF THE PRODUCT  
OF ASTM DENSITY AND SIZE RATINGS)

| ASTM Blister<br>Density<br>Rating*                                                                                                                                                                                                 | ASTM Blister Size Rating* |     |     |     |     |     |     |     |     |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------|-----|-----|-----|-----|-----|-----|-----|-----|
|                                                                                                                                                                                                                                    | No Blisters<br>(Size 10)  | 9   | 8   | 7   | 6   | 5   | 4   | 3   | 2   |
| No Blisters- 10                                                                                                                                                                                                                    | 10                        |     |     |     |     |     |     |     |     |
| Very Few - 9                                                                                                                                                                                                                       |                           | 9   | 8.5 | 7.9 | 7.3 | 6.7 | 6   | 5.2 | 4.2 |
| Few - 8                                                                                                                                                                                                                            |                           | 8.5 | 8   | 7.5 | 6.9 | 6.3 | 5.7 | 4.9 | 4   |
| - 7                                                                                                                                                                                                                                |                           | 7.9 | 7.5 | 7   | 6.5 | 5.9 | 5.3 | 4.6 | 3.7 |
| Medium - 6                                                                                                                                                                                                                         |                           | 7.3 | 6.9 | 6.5 | 6   | 5.5 | 4.9 | 4.2 | 3.5 |
| - 5                                                                                                                                                                                                                                |                           | 6.7 | 6.3 | 5.9 | 5.5 | 5   | 4.5 | 3.9 | 3.2 |
| Medium Dense - 4                                                                                                                                                                                                                   |                           | 6   | 5.7 | 5.3 | 4.9 | 4.5 | 4   | 3.5 | 2.8 |
| - 3                                                                                                                                                                                                                                |                           | 5.2 | 4.9 | 4.6 | 4.2 | 3.9 | 3.5 | 3   | 2.4 |
| Dense - 2                                                                                                                                                                                                                          |                           | 4.2 | 4   | 3.7 | 3.5 | 3.2 | 2.8 | 2.4 | 2   |
| * Based on use of ASTM D714 to evaluate blistering of coatings with respect to blister size and blister frequency (or density). The odd numbers in the density and size scales are interpolations between the values used in D714. |                           |     |     |     |     |     |     |     |     |

Information to be recorded during panel preparation is:

1. Panel identification number (front and back are separate samples)
2. Grit blast material used
3. Profile inspection results
4. Coating identification from paint can

5. Number and thickness of each coating (average of 5 dry film thickness measurements for each side)
6. Curing or drying conditions (in accordance with manufacturers specification)
7. Notes of any unusual, unexpected or pertinent factors or events

#### EXPOSURE APPARATUS

The exposure apparatus consists of the following items, as illustrated in Figure 1: 1) An exposure container made of corrosion-proof material such as glass, earthenware, porcelain-coated steel, or heat-resistant plastic. It shall have a clear transparent window to permit viewing and inspection of the panels without removing any panel from the water bath. It shall be large enough to accommodate the test panels with a spacing of not less than 13 mm (1/2 in.) between adjacent panels and with a space between the rows of panels so each can be removed with its hanger and brought to the inspection window while remaining immersed in the water. 2) An immersion-type bath circulator to heat and control the water temperature and to circulate the water to maintain uniform conditions around all the panels. 3) A source of deionized or distilled water so the water bath can be refreshed to maintain the conductivity at 20  $\mu$ mhos or less. A fill and drain system can be used to continuously change the water at a rate that permits the heater to maintain the desired temperature. Insulating the outside and covering the tanks will reduce the heat loss and water evaporation and provide better control of exposure conditions. Figure 1 shows a typical tank setup with the necessary components.

#### EXPOSURE PROCEDURE

The exposure procedure consists of the following steps:

1. Set up the tanks, fill with deionized water and heat to the desired temperature for at least 24 h before the panel exposure is to start to be sure the system is operating stably with a constant temperature and the specified conductivity.
2. Immerse the panels in the order they are to be inspected and rated. If large numbers of panels are to be exposed, the panels should be put into the water in small groups with a time schedule similar to the schedule required to rate the panels. In this way, the time of exposure of the first panels in the water before they are inspected will very nearly equal the time of exposure of the last panels before their first inspection.

3. Rate each panel at least once every 24 h for the duration of the exposure. The criterion for the frequency of rating must be based on the objective of the test. If the objective is to develop the time history curve of blistering, one needs 10 to 15 data points. For the 2-week exposure test, this works out to one rating per day. If the objective is to determine what coatings blister first or to rate the coatings based on time to blister initiation, the ratings must be more frequent to ensure that differences in time to blistering can be determined to the precision desired.

4. Inspect each panel through the front glass. If the hot panels are taken from the 180°F water into ambient temperature air, the thermal shock will almost certainly affect the coating and the test in negative and uncontrolled ways. To make the raised blister more visible a spot light can be directed on the panel through the glass at an angle to highlight the blisters.

5. The condition of the panel is rated using ASTM D-714, "Evaluating Degree of Blistering of Paints," to obtain quantitative values. The ASTM D-714 ratings are converted into the single numerical "linear blister resistances rating" by using Table 1.

6. Each rating of a panel must be made without reference to previous ratings of that same panel. This method preserves the independence of the ratings and prevents bias in later readings. The spread of the data will naturally be somewhat larger but the averages will more truly represent the actual conditions.

7. If more than one person is rating the panels, they can alternate taking readings or they can independently rate the panels and report the average of their ratings for each rating period. Before any rating is made the investigators doing the rating must agree on what density ratings are to be used for panels that have only one, two, or three blisters in the rating area. (These ratings are not specified in the ASTM D-714.) It is recommended that one blister be rated as 9.9 for density or frequency, and two blisters be rated as 9.5 for density or frequency. For three or more the D-714 scheme can be used.

8. Record for each rating session: Time, date, panel number, density or frequency, size, and any comments that pertain to the ratings. In addition the conductivity of the water in the tanks should be measured daily and recorded. If the conductivity goes above 20  $\mu$ mhos, the water should be refreshed, carefully to keeping the temperature within requirements while deionized water is being slowly added.

## DISCUSSION

Some representative results of actual blister tests are given in Figures 2, 3 and 4.

Note that these figures do not include a criterion to define failure of the coating by blistering. What constitutes failure must be defined by the user of the data based on his or her requirements. However, given that these paints are expected to perform from 4 to 7 years in service, it is reasonable to expect very few or no blisters in the hot, deionized water exposure for only 2 weeks. In recent tests, 10% of MIL-P-24441 coatings and 20% of the commercial AC coatings had an average blister resistance rating of 9.0 or better at the end of the 2-week exposure to hot, deionized water. These resistance ratings for each coating were the averages of 96 independent ratings (8 ratings for each of 12 panel sides over the last 84 h of the 336-h test). Thus, there are anticorrosive coatings available that will pass the blister test described here when failure is defined as very few or no blisters.

If a blister resistance rating of 9.0 is considered too stiff a criterion, the data are available to select any other value. Alternatively, one may simply rank the tested coatings at the end of the 2-week exposure test.

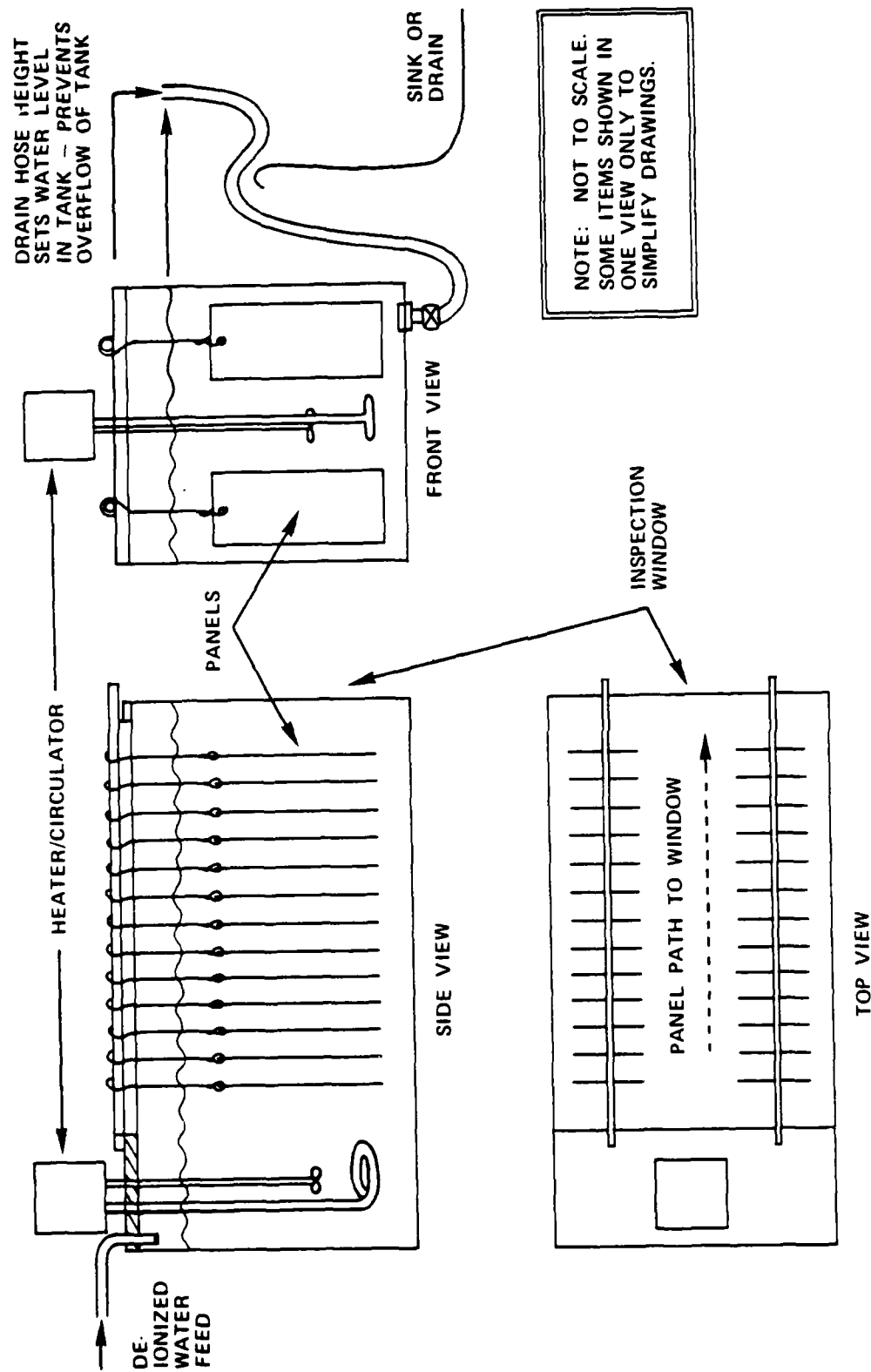


Figure 1 - Schematic of Exposure Tank Arrangement (Specific tank arrangement will depend on equipment available)

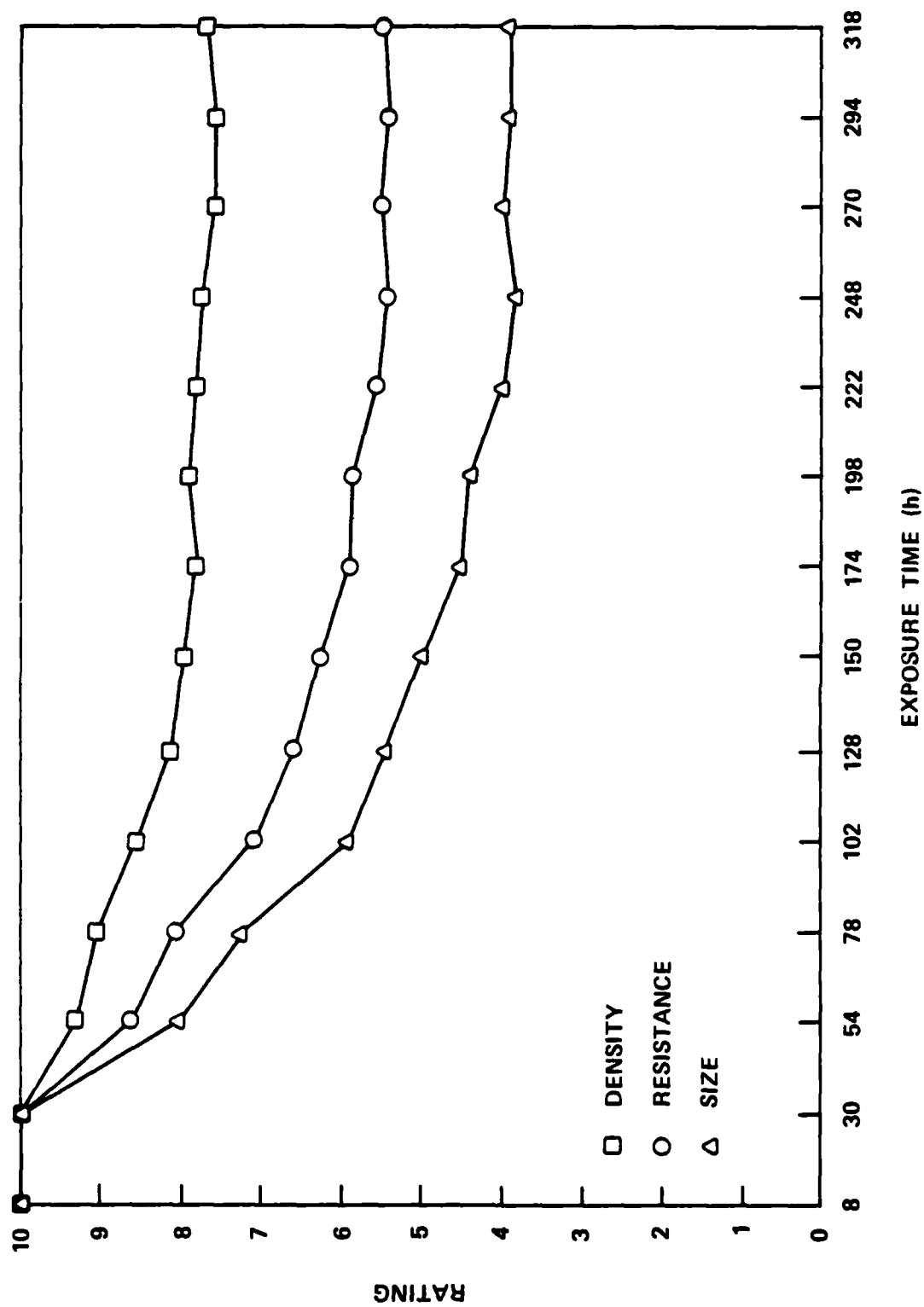


Figure 2 - Typical Blister Test Results (Paint No. 1)

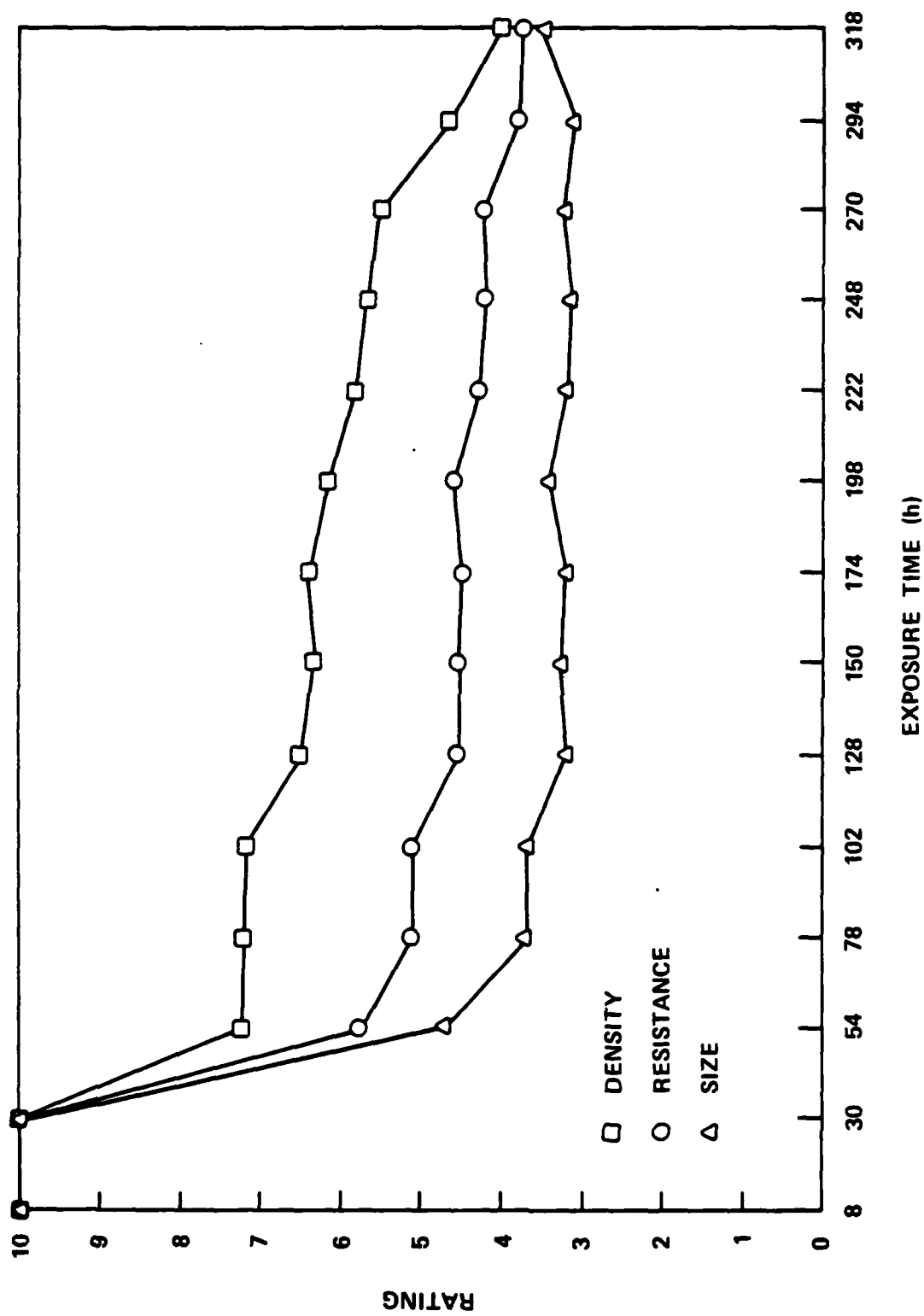


Figure 3 - Typical Blister Test Results (Paint No. 29)



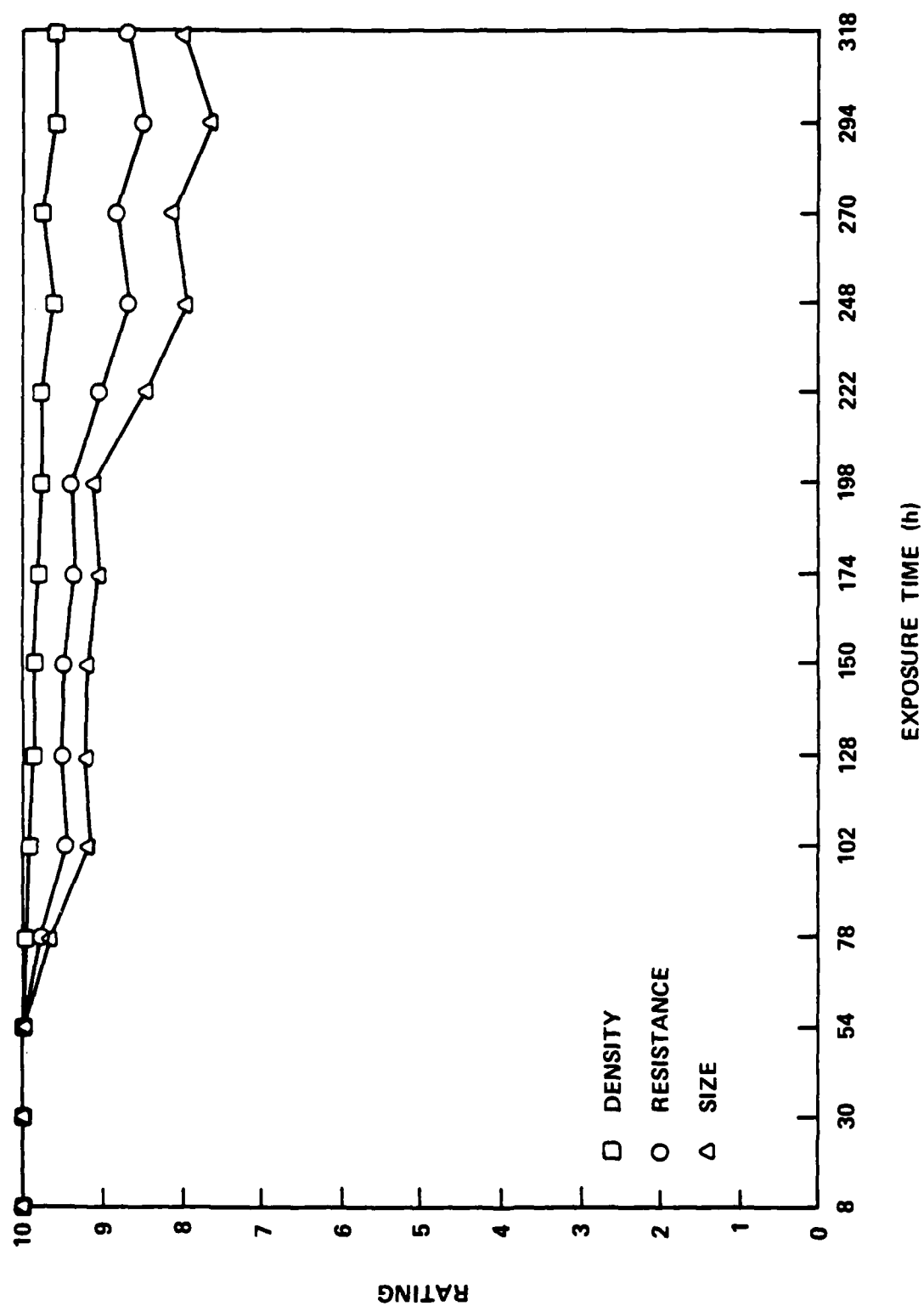


Figure 4 - Typical Blister Test Results (Paint No. 10)

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